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# MECHANICAL PROPERTIES OF DILUTE TUNGSTEN-RHENIUM ALLOYS\*

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## SUMMARY

Tungsten alloys containing 1.9 to 9.1 weight percent rhenium and 1 to 7 percent rhenium were prepared by electron-beam and arc melting, respectively. These were warm fabricated into sheet and/or rod and evaluated by low-temperature bend and tensile studies, high-temperature tensile and creep studies, and recrystallization and grain growth studies. A commercial arc-melted 26-percent-rhenium alloy and an electron-beam-melted 24-percent-rhenium alloy were also evaluated for comparison.

The dilute tungsten-rhenium alloys were significantly more ductile than unalloyed tungsten fabricated in a similar manner. Ductile-brittle bend transition temperatures of  $-75^{\circ}$  and  $-100^{\circ}$  F were observed for worked sheet of the electron-beam-melted alloys with 1.9 and 9.1 percent rhenium, respectively. The dilute arc-melted alloys were slightly less ductile than the electron-beam-melted alloys, and room-temperature ductility was observed only with the 1.0 percent rhenium alloy. These compared with transition temperatures of  $215^{\circ}$  and  $235^{\circ}$  F for worked sheet of unalloyed arc- and electron-beam-melted tungsten, respectively. Transition temperatures for the arc-melted 26-percent-rhenium alloy and the electron-beam-melted 24-percent-rhenium alloy were  $-150^{\circ}$  and approximately  $-310^{\circ}$  F, respectively.

Annealing at  $3600^{\circ}$  F recrystallized all the alloys and significantly increased the bend transition temperatures. Minimums in transition temperature of  $450^{\circ}$  and  $400^{\circ}$  F occurred at 2 percent rhenium in the series of electron-beam-melted alloys and at about 4 percent rhenium in the arc-melted alloys, respectively, compared with  $630^{\circ}$  to  $670^{\circ}$  F for unalloyed tungsten. The 24- and 26-percent-rhenium alloys, with transition temperatures of  $375^{\circ}$  and  $350^{\circ}$  F, respectively, were slightly more ductile than the best dilute alloys after this anneal.

At  $2500^{\circ}$  to  $3500^{\circ}$  F, the short-time tensile strength increased with increasing rhenium content up to 9.1 percent rhenium.

The creep strength at  $3500^{\circ}$  F increased with increasing rhenium content up to 5 to 7 percent rhenium. The 24- and 26-percent-rhenium alloys were weaker than the dilute alloys and had about the same creep strength as unalloyed tungsten.

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\* Portions of the study presented herein were presented at the AIME Technical Conference on Physical Metallurgy of Refractory Metals, French Lick, Indiana, October 1-3, 1965, in a paper entitled "Ductility and Strength of Dilute Tungsten-Rhenium Alloys."

## INTRODUCTION

Recent studies have shown that the high-temperature strength of tungsten can be significantly increased by alloying; however, the lack of ductility in these materials at ambient temperatures remains a deterrent to their future use. Various approaches, including purification and alloying, have been employed to alleviate the ductility problem.

Consolidation by electron-beam melting improves the purity with respect to trace metallics, as compared with arc melting or powder metallurgy techniques. The unalloyed electron-beam-melted tungsten, however, does not possess improved low-temperature ductility (ref. 1) except in the form of fine wire (ref. 2).

The extraordinary effects of high-rhenium additions (in the range 22 to 39 atomic percent) in promoting low-temperature ductility in tungsten, molybdenum, and chromium are well known (refs. 3 and 4), although a satisfactory description of the mechanism of this effect has not yet evolved. The observation of a significant decrease in hardness (8 to 10 percent) on the addition of about 5 percent rhenium to tungsten has prompted several studies to determine if improved ductility could be achieved in these dilute, less costly, tungsten-rhenium alloys. Pugh et al. (ref. 5) have demonstrated that this is the case with fine wire of doped tungsten made by powder metallurgy techniques. In a previous study (ref. 6), room-temperature bend ductility in worked sheet fabricated from electron-beam-melted tungsten alloys with 2 and 6 percent rhenium was observed.

Because of the desirability of improving the low-temperature ductility of tungsten, the work begun in reference 6 was extended to determine the extent of the ductility improvement and to characterize the effects of composition and melting method on ductility and other properties of the alloys.

## EXPERIMENTAL PROCEDURES

### Materials

The materials consisted of -325 mesh, commercially pure (undoped) tungsten powder and -200 mesh, commercially pure rhenium powder. Electrodes that measured nominally  $1\frac{1}{8}$  inches in diameter by 24 inches long were compacted from the blended powders and consolidated either by triple electron-beam melting or by arc melting. The ingots were  $2\frac{1}{2}$  inches in diameter and ranged from about 4 to 8 inches in length.

The compositions selected included eight electron-beam-melted and five arc-melted dilute alloys with 1.0 to 9.1 percent rhenium. In addition, an electron-beam-melted tungsten - 24-percent-rhenium alloy and an arc-melted commercial tungsten - 26-percent-rhenium alloy were included for comparison with the dilute alloys. Alloys were single

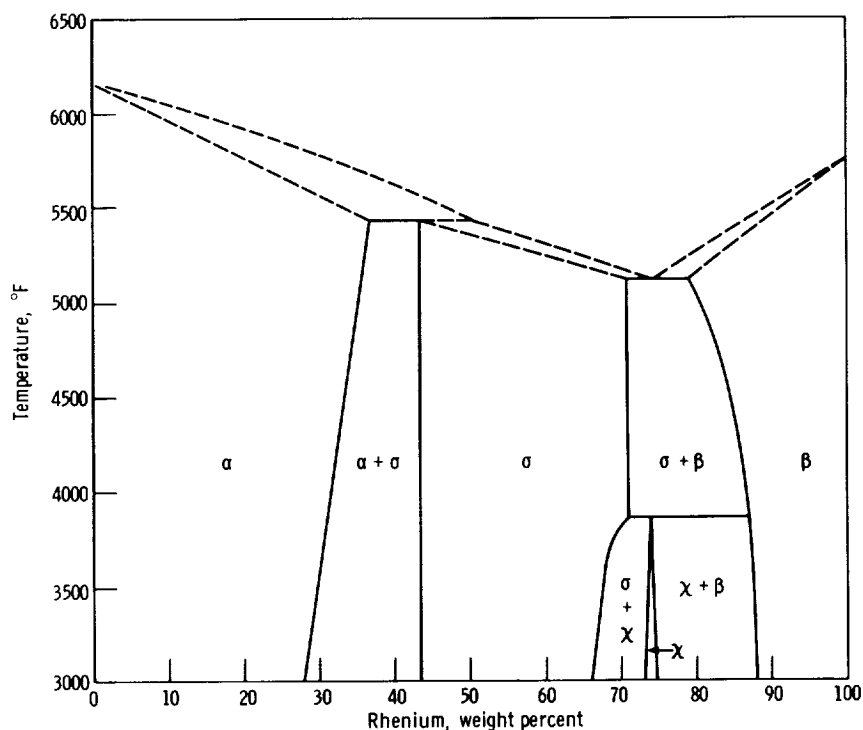


Figure 1. - Tungsten-rhenium phase diagram (ref. 7).

phased, in agreement with the phase diagram shown in figure 1 (ref. 7). Chemical analyses of the alloys are given in table I.

## Fabrication

The fabrication details for both the electron-beam- and arc-melted ingots are summarized in table II. Two extrusion billets were obtained from each of five electron-beam-melted ingots, while one billet was machined from each of the other four electron-beam-melted ingots and from each of the five arc-melted ingots.

The electron-beam-melted ingots were machined into billets measuring 1.75 inches in diameter by 4.06 inches long. These were canned in 3/16-inch-wall powder-metallurgy unalloyed molybdenum for extrusion. The arc-melted ingots were machined into 2.25-inch-diameter billets about 4 inches long and canned in 3/8-inch-wall molybdenum. The billets were extruded in a hydraulic extrusion press at temperatures ranging from 3200° to 4200° F. Nine of the electron-beam-melted billets were extruded into sheet bar, while the other five electron-beam-melted billets and all five arc-melted billets were extruded to rounds. The reduction ratios were 6 or 8, as shown in table II. No difficulties were encountered with any of the 19 extrusions.

Rod and sheet were fabricated from the extrusions, as indicated in table II. Fabri-

TABLE I. - ANALYSES AND HARDNESSES OF ALLOY INGOTS

Element	Ingot															
	Electron-beam-melted alloy								Arc-melted alloy							
	EB-160	EB-127	EB-156	EB-159	EB-139	EB-176	EB-126	EB-179	EB-181	A-132	A-134	A-102	A-99	A-138	(a)	
Nominal rhenium, weight percent																
2	3	3	3	4	5	5	7	10	26	1	2	3	5	7	25	
Analyzed rhenium, weight percent																
1.9	2.5	2.8	3.6	4.5	4.7	6.5	9.1	24	1.0	2.0	3.0	5.1	6.8	26		
Vickers hardness																
370	326	323	322	314	312	297	314	417	366	345	353	313	307	--		
Impurity content, ppm																
Oxygen	10	2	3	2	3	4	5	1	8	3	2	2	3	12	28	
Nitrogen	<3	<5	<3	<3	<5	<3	<5	<3	<3	5	5	5	46	<5	29	
Carbon	4	5	5	3	2	4	8	4	4	3	4	2	7	3	44	
Aluminum	--	--	0.02	--	--	0.01	--	0.1	--	0.06	--	0.05	0.03	0.1	<10	
Beryllium	--	--	.02	--	--	.02	--	.003	--	.002	--	.002	.001	.009	---	
Calcium	--	--	.02	--	--	.014	--	.1	--	.03	--	.03	<.01	.03	---	
Cobalt	--	--	.02	--	--	.02	--	<.01	--	.02	--	.2	.02	.04	<5	
Chromium	--	--	1.1	--	--	2.3	--	2.2	--	.6	--	1.4	1.9	.5	<10	
Copper	--	--	.007	--	--	.01	--	.01	--	.02	--	.002	.001	.004	1	
Iron	--	--	.14	--	--	.2	--	.5	--	1.6	--	1.8	6.1	1.2	17	
Magnesium	--	--	.001	--	--	.001	--	.001	--	.001	--	.001	<.001	.001	<1	
Manganese	--	--	.004	--	--	.002	--	.004	--	.01	--	.01	.01	.01	<10	
Molybdenum	--	--	53	--	--	77	--	95	--	31	--	39	16	48	78	
Niobium	--	--	.3	--	--	.2	--	.3	--	40	--	15	35	75	---	
Nickel	--	--	.15	--	--	.1	--	.1	--	.3	--	.3	.8	.2	1	
Silicon	--	--	.3	--	--	.2	--	.2	--	.3	--	.2	.2	.3	<20	
Tantalum	--	--	45	--	--	5	--	2	--	100	--	24	81	33	---	
Titanium	--	--	.01	--	--	<.01	--	.02	--	.04	--	.04	.02	.02	<1	
Vanadium	--	--	.04	--	--	.03	--	.03	--	.15	--	.13	.09	.14	<10	
Zirconium	--	--	.05	--	--	<.01	--	.01	--	1.6	--	>10	<.01	1.3	---	

<sup>a</sup>Supplier's analysis; obtained commercially.



TABLE II. - FABRICATION OF MATERIALS

Ingot	Analyzed rhenium content, wt percent	Extrusion			Swaging		Rolling					
		Tempera- ture, °F	Reduc- tion ratio	Type	Temperature, °F	Reduc- tion, percent	Temperature, °F	Inter- mediate condi- tion- ing <sup>a</sup>	Inter- mediate clean- ing <sup>b</sup>	Reduc- tion, percent	Final thick- ness, in.	Bend transi- tion tem- perature, as rolled, °F
Electron-beam-melted alloys												
EB-160A	1.9	3400	6	Round	2300 to 2100	76	-----	-	-	--	----	----
EB-160B	1.9	3200	8	Sheet	-----	--	2350 to 2000	3	3	93	0.030	-75
EB-127	2.5	3400	6	Round	2600 to 2250	76	2580 to 1750 2580 to 2000	1 1	0 0	94 94	0.051 .050	25 ≤75
EB-156A	2.8	3400	8	Sheet	-----	--	2400 to 2400 2400 to 2600 2400 to <sup>c</sup> 2000 2400 to <sup>c</sup> 2200	1 ↓	0 ↓	91 ↓	0.030 ↓	≤75 ≤50 25 0
EB-156B	2.8	3200	8	Sheet	-----	--	2400 to 2000 2400 to 2200 2400 to <sup>c</sup> 2200	1 1 1	0 0 0	91 91 91	0.030 .030 .030	-25 50 0
EB-159A	3.6	3400	6	Round	2300 to 2100	76	-----	-	-	--	----	----
EB-139A	4.5	3400	6	Round	2550 to 2200	76	-----	-	-	--	----	----
EB-139B	4.5	3400	8	Sheet	-----	--	2450 to 1800 2450 to 2000 2450 to 2200	1 1 1	0 0 0	91 91 91	0.030 .030 .030	-25 -25 50
EB-176A	4.7	3400	8	Sheet	-----	--	2350 to 2000	3	3	93	0.030	-75
EB-176B	4.7	3400	8	Sheet	-----	--	2800 to 2600	3	3	93	0.030	175
EB-126	6.5	3400	8	Round	2550 to 2250	68	2600 to 2000 2620 to 2150	1 1	0 0	92 92	0.049 .053	25 25
EB-179	9.1	3500	6	Sheet	-----	--	2350 to 2000	3	3	93	0.030	-100
EB-181	24	4200	6	Sheet	-----	--	2350 to 2600	3	3	94	0.030	≤-310
Arc-melted alloys												
A-132	1.0	3600	8	Round	2500 to 2280	81	2450 to 2225	1	0	89	0.049	75
A-134	2.0	3800	8	Round	2600 to 2450	81	-----	-	-	--	----	----
A-102	3.0	4000	8	Round	-----	--	2720 to 2370	1	0	92	0.048	280
A-99	5.1	4000	8	Round	-----	--	2760 to 2410	1	0	92	0.051	240
A-138	6.8	4000	8	Round	2800 to 2600	81	2750 to 2700	1	0	89	0.050	50

<sup>a</sup>Grinding out of surface defects.<sup>b</sup>Salt-bath cleaning to remove surface oxide.<sup>c</sup>Sheets given intermediate anneals for 1/2 hour at 2200° F in hydrogen at thicknesses of 0.070 and 0.045 inch.

cation temperatures for the arc-melted alloys were slightly higher than those for the electron-beam-melted alloys. The structures are comparable, however, since arc-melted tungsten has a higher recrystallization temperature than electron-beam-melted tungsten (ref. 1).

Several variations in the sheet rolling procedures were introduced in order to evaluate their effects on the sheet bend ductility of the electron-beam-melted alloys. Final rolling temperature was varied between 1750° and 2600° F, intermediate stress-relief anneals were introduced, and the extent of conditioning (grinding of defects) and salt-bath cleaning during fabrication was varied.

## Bend Testing

Bend test specimens measuring 0.3 by 0.9 inch were cut from the rolled alloy sheets by using a cutoff wheel. Larger specimens measuring 0.5 by 2 inches were also cut from the electron-beam-melted 2.8-percent-rhenium alloy so that the effect of specimen size on bend ductility could be checked. All the specimens were electropolished in a 2-percent-aqueous sodium hydroxide solution to remove 3 to 5 mils of metal per side before bend testing, except those specimens evaluated after salt-bath cleaning only. The electropolishing process removes surface flaws and improves the reproducibility of bend testing. Heat treatment of the specimens prior to bend testing was conducted in an induction-heated hydrogen atmosphere tube furnace (1800° to 3200° F) or in a vacuum ( $10^{-5}$  torr) with a resistance-heated tungsten element (3400° to 4200° F). Temperatures were measured in both furnaces by tungsten - tungsten-25-percent-rhenium thermocouples. Bend tests were performed at a crosshead speed of 1 inch per minute over a bend radius of four times the specimen thickness 4T. A controlled liquid-nitrogen spray was employed for tests at -25° F and lower, while dry-ice - acetone mixtures were used to obtain temperatures between -25° and 75° F. For temperatures between 75° and 800° F, a resistance-heated air-atmosphere tube furnace was employed.

The bend test apparatus used in the different temperature ranges varied slightly but was characterized in every case by rollers for the plunger and the two support points. The bend apparatus used at 75° F and higher is shown in detail in reference 8. The bend transition temperature is defined as the lowest temperature at which a specimen could be bent 90° without fracture.

## Tensile and Creep Testing

Sheet and/or rod specimens were machined from each alloy to study the low- and

high-temperature tensile properties and high-temperature creep properties. Sheet specimens had a 0.25-inch-wide by 1-inch-long reduced section. The grip ends were pinned to prevent slippage during testing. Rod specimens were machined with a 0.16-inch-diameter by 1.03-inch-long reduced section.

The low-temperature tensile and ductility properties were studied on rod and sheet specimens that were electropolished prior to testing to remove 3 to 5 mils from each surface. The crosshead separation rate was 0.005 inch per minute until about 0.5 percent plastic strain, after which the rate was increased to 0.05 inch per minute.

Tensile tests at 2500<sup>o</sup> to 4000<sup>o</sup> F were conducted in a water-cooled stainless steel vacuum unit ( $1 \times 10^{-5}$  torr) equipped with a tubular tantalum sleeve heater. This unit is described in reference 9. Crosshead speed was 0.05 inch per minute throughout the test.

Step-load creep tests were conducted in a conventional beam-load unit that was equipped with a water-cooled vacuum shell and a tantalum heater similar to that used for tensile testing. Specimen extensions were measured from load rod motion by a dial indicator. A comparison of steady creep rates calculated from loading rod movement with those calculated from absolute strain measurements from optical cathetometer readings indicated almost identical creep rates. The strains, however, from loading rod movement were consistently 1 to 2 percent greater than the values from cathetometer readings; this difference reflected creep of the load train and settling of the grips during initial loading.

## Recrystallization and Grain Growth Studies

Recrystallization studies were conducted on sheet and rod from the electron-beam-melted alloys in order to determine the temperature for 50-percent recrystallization in 1 hour and grain growth characteristics after recrystallization. Specimens were heated for 1 hour at 2400<sup>o</sup> to 4000<sup>o</sup> F and metallographically examined. The extent of recrystallization was averaged from visual estimates by two observers over at least 10 areas on each specimen. Grain sizes on fully recrystallized specimens were determined by the line-intercept method (ref. 10).

## RESULTS AND DISCUSSION

### Ductile-Brittle Bend Transition Behavior

Effects of composition. - The bend transition temperatures determined for the electron-beam- and arc-melted alloys as rolled and after heat treating at 1800<sup>o</sup> to 4200<sup>o</sup> F are summarized in table III. Figure 2 shows the bend transition temperatures as rolled

TABLE III. - BEND TRANSITION TEMPERATURES FOR TUNGSTEN - RHENIUM ALLOYS

Fabrication schedule		1-Hour annealing temperature, °F	Average grain diameter, in.	Bend transition temperature, °F	Fabrication schedule		1-Hour annealing temperature, °F	Average grain diameter, in.	Bend transition temperature, °F
Temperature, °F	Reduction, percent				Temperature, °F	Reduction, percent			
EB-160B, tungsten - 1.9 percent rhenium					EB-176, tungsten - 4.7 percent rhenium				
2000	93	As rolled	(a)	-75	2000	93	As rolled	(a)	-75
		1800		0			2200	(a)	0
		2000		350			2400	(a)	275
		2200		-25			2600	(b)	300
		2400	(b)	350	2600	93	As rolled	(a)	175
		2600	0.00098	375			2200	(a)	125
		3000	c. 0010	475			3000	-----	400
		3600	.0051	425			3600	0.0029	675
EB-127, tungsten - 2.5 percent rhenium					EB-126, tungsten - 6.5 percent rhenium				
1750	94	As rolled	(a)	25	2000	92	As rolled	(a)	25
		2200		30			2200	(a)	100
		2400		150			3000	0.00091	475
		2600		225			3600	c. 0028	775
		3000	0.0011	500			4200	c. 010	850
2000	94	As rolled	(a)	≤75	2150	92	As rolled	(a)	25
		2200	(a)	≤75			2200	(a)	25
		3000	0.0011	325			3200	0.0010	385
		3600	.0034	500			3600	c. 0028	700
		4200	c. 012	725			EB-179, tungsten - 9.1 percent rhenium		
EB-156A, tungsten - 2.8 percent rhenium					2000	93	As rolled	(a)	-100
d <sub>2200</sub>	91	As rolled	(a)	0			1800		0
		1800		75			2200		150
		2000		-25			2400		125
		2200		-25			2600	(b)	225
		2400		50			3000	c. 00083	250
		2600		200			3600	.0020	750
		3600	0.0033	450			EB-181, tungsten - 24 percent rhenium		
EB-139B, tungsten - 4.5 percent rhenium					2600	94	As rolled	(a)	≤-310
1800	91	As rolled	(a)	-25			1800		-225
		2000	(a)	0			2000		-250
		2200	(a)	50			2200		-250
		3600	0.0031	625			2400		-225
2000	91	As rolled	(a)	-25			2600	(b)	50
		2200	(a)	≤25			3000	c. 00095	75
		4200	-----	525	3600	.0026	375		
2200	91	As rolled	(a)	50	A-132, tungsten - 1.0 percent rhenium				
		1800		≤50	2225	89	As rolled	(a)	75
		2000		25			2200		150
		2200		25			2400		75
		2400		≤-25			2600		225
		2600		25			3000		425
		2800	0.0010	400			3600	0.0015	425
		3600	.0024	500			4200	.0043	575

<sup>a</sup>Worked microstructure.<sup>b</sup>Partially recrystallized microstructure.<sup>c</sup>Grain size estimated from other data on same alloys (see table IX).<sup>d</sup>This sheet given intermediate stress relief anneals for 1/2 hr at 2200° F in hydrogen at thicknesses of 0.070 and 0.045 in.

TABLE III. - CONCLUDED. BEND TRANSITION  
TEMPERATURE FOR TUNGSTEN -  
RHENIUM ALLOYS

Fabrication schedule		1-Hour annealing temperature, °F	Average grain diameter, in.	Bend transition temperature, °F
Temperature, °F	Reduction, percent			
A-102, tungsten - 3.0 percent rhenium				
2370	92	As rolled	(a)	280
		2200	(a)	280
		2600	(a)	<200
		3600	0.0010	400
A-99, tungsten - 5.1 percent rhenium				
2410	92	As rolled	(a)	240
		2200	(a)	255
		2600	(a)	100
		3600	0.00083	375
		4200	-----	725
A-138, tungsten - 6.8 percent rhenium				
2700	89	As rolled	(a)	50
		3600	-----	475
Tungsten - 26 percent rhenium				
----	--	As received	(a)	-150
		3000	0.00079	75
		3600	.0022	350
		4200	.0075	500

<sup>a</sup>Worked microstructure.

and after annealing at 3000° and 3600° F. The data plotted in figure 2(a) are for the lowest rolling temperatures studied (on the electron-beam-melted alloys) and represent the lowest transition temperatures observed in this study. Dilute rhenium alloying effects a significant improvement in the low-temperature ductility of electron-beam-melted tungsten, particularly in the worked condition (fig. 2(a)). These alloys exhibit ductile-brittle transition temperatures as low as -100° F. All the electron-beam-melted alloys had ductile-brittle transition temperatures of 25° F or lower in the best condition evaluated.

As seen in figure 2, rhenium is effective at low alloying levels. The electron-beam-melted tungsten - 1.9-percent-rhenium alloy showed a transition temperature of -75° F as rolled and 425° F after annealing at 3600° F, both of which represent 200° to 300° F improvement over unalloyed tungsten (ref. 1). The low transition temperature in the worked condition is particularly significant, since it suggests that damage during handling can be reduced, and

some slow forming operations on this material can be conducted at ambient temperatures. Outstanding ductility was also exhibited by the as-rolled electron-beam-melted tungsten - 9.1-percent-rhenium alloy with a transition temperature of -100° F.

The intermediate alloys, which contain 2.5 to 6.5 percent rhenium, were slightly less ductile, with transition temperatures ranging between 25° and -25° F. It is believed that the ductility of these as-rolled alloys is significantly affected by cleaning and rolling conditions and resulting structures, as is discussed in more detail on page 13. The very low transition temperatures of -75° and -100° F may represent the best ductilities obtainable in this composition range, while the higher transition temperatures are probably associated with less than optimum structures or cleaning procedures.

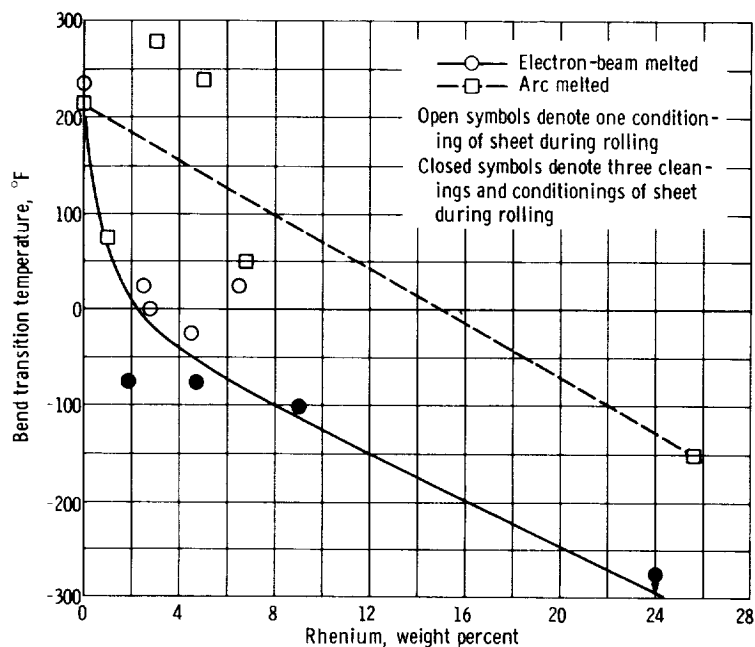
In comparison to the electron-beam-melted alloys, the arc-melted alloys were less ductile, with transition temperatures ranging between 50° and 280° F in the as-rolled condition. Considerable scatter was also observed in the transition temperatures for the as-rolled arc-melted materials. The arc-melted alloys with 3 and 5 percent rhenium were prepared in a deep mold configuration, whereas the remaining arc-melted alloys

were melted near the top of the mold using a retractable stool. The latter two alloys are most likely of slightly higher purities. Also, as pointed out on page 3, the fabrication procedure for these alloys differed slightly from the electron-beam-melted alloys and was not optimized to the same extent. Some of the indicated differences in ductility between the arc- and electron-beam-melted alloys may be attributable to this factor.

As expected, the high-rhenium alloys showed excellent ductility in the as-rolled condition. The bend transition temperature of the electron-beam-melted tungsten - 24-percent-rhenium alloy, less than  $-275^{\circ}\text{F}$ , was lower than that of the worked (as-received) arc-melted tungsten - 26-percent-rhenium alloy,  $-150^{\circ}\text{F}$ . Rhenium alloying is also effective in improving the ductility of fully recrystallized materials, as shown in figures 2(b) and (c).

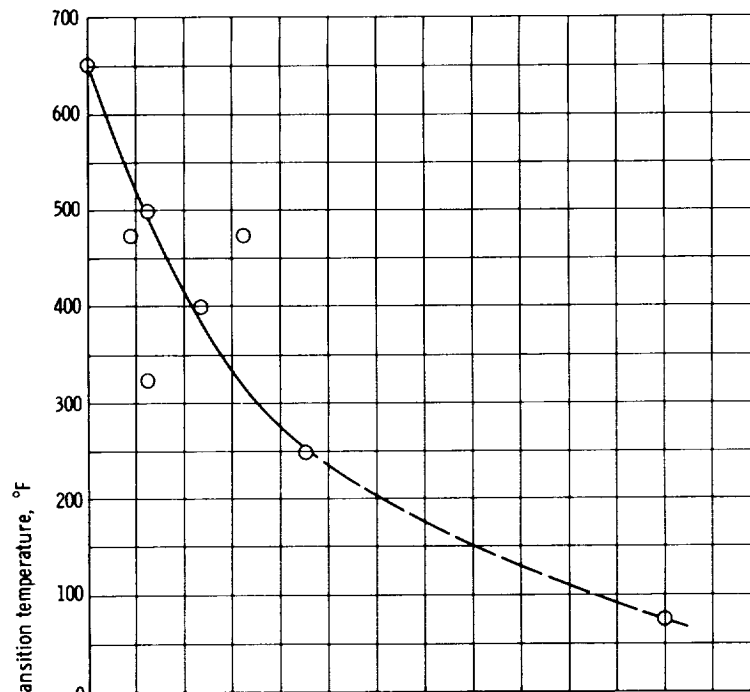
These data indicate that rhenium improves the ductility of materials annealed at  $3000^{\circ}\text{F}$ . This behavior is similar to that observed for the as-rolled alloys (fig. 2(a)). After annealing at  $3600^{\circ}\text{F}$ , minimums in the transition temperature-composition curves were observed at about 2 percent rhenium for the electron-beam-melted alloys and at about 4 percent rhenium for the arc-melted alloys. At slightly higher rhenium levels, the transition temperatures for the electron-beam-melted alloys are higher than that for unalloyed tungsten. The  $3600^{\circ}\text{F}$  recrystallized arc-melted alloys have lower transition temperatures than the electron-beam-melted alloys.

The high-rhenium alloys, which deform initially by twinning in the recrystallized condition, were only slightly more ductile than the dilute alloys, which deform entirely

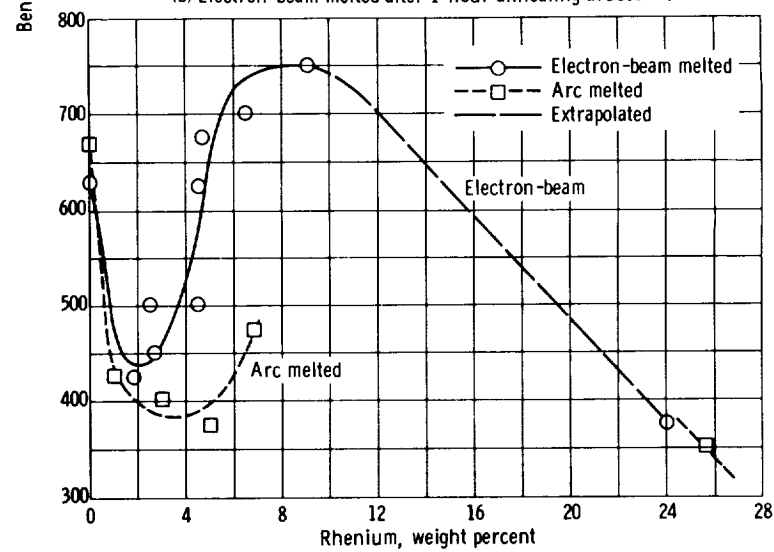


(a) Electron-beam and arc melted, as rolled.

Figure 2. - Bend transition temperatures of as-rolled tungsten-rhenium alloys.

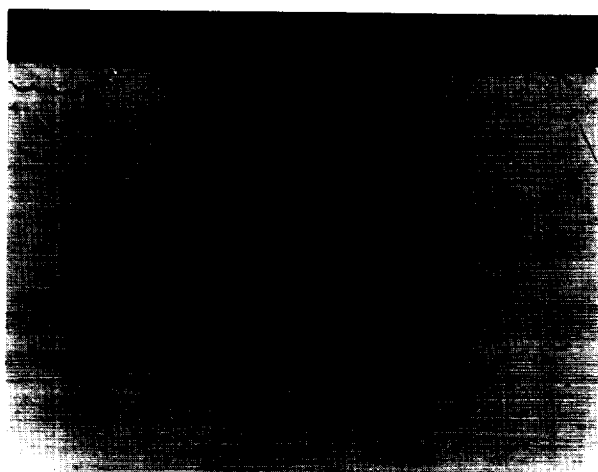


(b) Electron-beam melted after 1-hour annealing at 3000° F.



(c) Electron-beam- and arc-melted after 1-hour annealing at 3600° F.

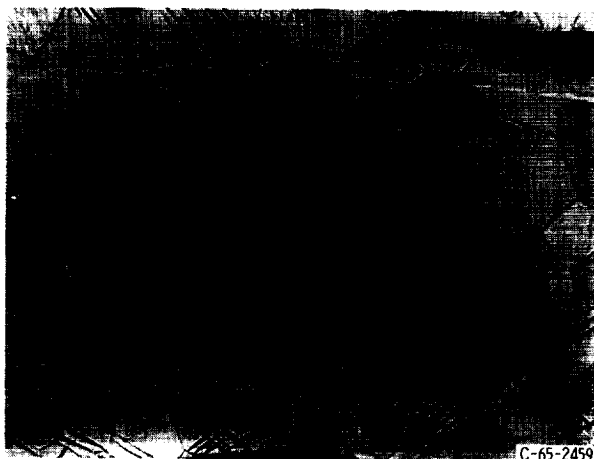
Figure 2. - Concluded.



(a) Tungsten - 2.5 percent rhenium, rolled at 1750° F, annealed at 2000° F, and bent to fracture at 25° F.



(b) Tungsten - 2.8 percent rhenium, annealed at 3600° F and bent to fracture at 400° F.



(c) Tungsten - 24 percent rhenium, annealed at 3600° F and bent to fracture at 300° F.

Figure 3. - Representative microstructures of electron-beam-melted tungsten-rhenium alloys. X150. (Reduced 50 percent in printing.)

by slip, after annealing at 3600° F. Transition temperatures of the high-rhenium alloys were 350° and 375° F compared with minimum of 375° and 425° F for the dilute alloys.

Purity and grain size appear responsible for the differences in ductility between the electron-beam- and arc-melted alloys. In the worked condition, the better ductility of the electron-beam-melted alloys is attributed to their higher purities. Although the analytical data in table I (p. 4) indicate little difference in either interstitial or metallic impurity contents between the electron-beam- and arc-melted alloys (with the exception of iron), it was shown in reference 1 that unalloyed electron-beam-melted tungsten is lower in metallic impurities than arc-melted tungsten. Further, as discussed on page 30, the grain sizes of the electron-beam-melted alloys after annealing at 3600° F were larger than those of the arc-melted alloys; this indicates higher grain growth rates and higher purity.

In the recrystallized condition, the transition temperatures of the tungsten-rhenium alloys are affected by grain size to a greater extent than is unalloyed tungsten, the larger grained materials having the higher transition temperatures. Thus, the larger grain sizes of the electron-beam-melted alloys apparently contribute to their higher transition temperatures as compared with arc-melted alloys after similar annealing treatments.



Representative microstructures of selected alloys after bending are shown in figure 3. Figure 3(a) illustrates crack propagation in a worked tungsten - 2.5-percent-rhenium specimen bent to fracture just below the transition temperature. A crack has encountered a plane of weakness in the sheet and temporarily changed its direction of propagation from transverse to longitudinal. This behavior produces the fibrous or laminated type of fracture that is characteristic of worked unalloyed tungsten also.

In figure 3(b), a transverse crack in a fully recrystallized specimen of tungsten - 2.8 percent rhenium is propagating partly intergranularly and partly transgranularly. Recent fractographic studies (private communication from A. Gilbert, Battelle Memorial Institute) indicated that the mode of crack propagation is about 50 percent transgranular in these alloys. This is in contrast to unalloyed, recrystallized electron-beam-melted tungsten, which fractures almost entirely intergranularly. The area shown in figure 3(b) is near final bend fracture.

The structure of a recrystallized specimen of tungsten - 24 percent rhenium adjacent to the fracture is shown in figure 3(c). This specimen exhibits profuse twinning, which is associated with cold deformation in the bend area and is characteristic of the high-rhenium alloys.

Effects of annealing. - The effects of annealing and recrystallization on the ductile brittle transition temperature are shown in figure 4 for unalloyed tungsten (refs. 1 and 9) and selected binary tungsten-rhenium alloys.

These plots indicate that unalloyed tungsten and alloys with 1.0 or 1.9 percent rhenium show a rather sharp increase in transition temperature on recrystallization, but the transition temperatures increase only slightly as the annealing temperature is increased further. In contrast, the alloys containing 5.1 to 26 percent rhenium show little change in transition temperature as they recrystallize; they rather exhibit a gradual increase in transition temperature with increasing annealing temperature.

It is tentatively concluded that the addition of moderate to high amounts of rhenium to tungsten, that is, about 5 to 26 percent, decreases the effect of recrystallization on the transition temperature. However, it also increases the effect of grain size; the coarser-grained structures that result from higher annealing temperatures have the higher transition temperatures.

Effects of fabrication variables. - The effects of rolling temperature, in-process anneals, salt-bath cleaning, pack rolling, and specimen size on the ductile-brittle transition temperature in the as-rolled condition were evaluated. These evaluations were conducted on the electron-beam-melted alloys since both previous work (ref. 6) and initial results from the present study showed that the electron-beam-melted alloys have lower transition temperatures than the arc-melted alloys.

Rolling temperatures between 1750<sup>0</sup> and 2600<sup>0</sup> F of the electron-beam-melted alloys with 2.5, 4.5, 4.7, and 6.5 percent rhenium were studied. As shown by table III, roll-

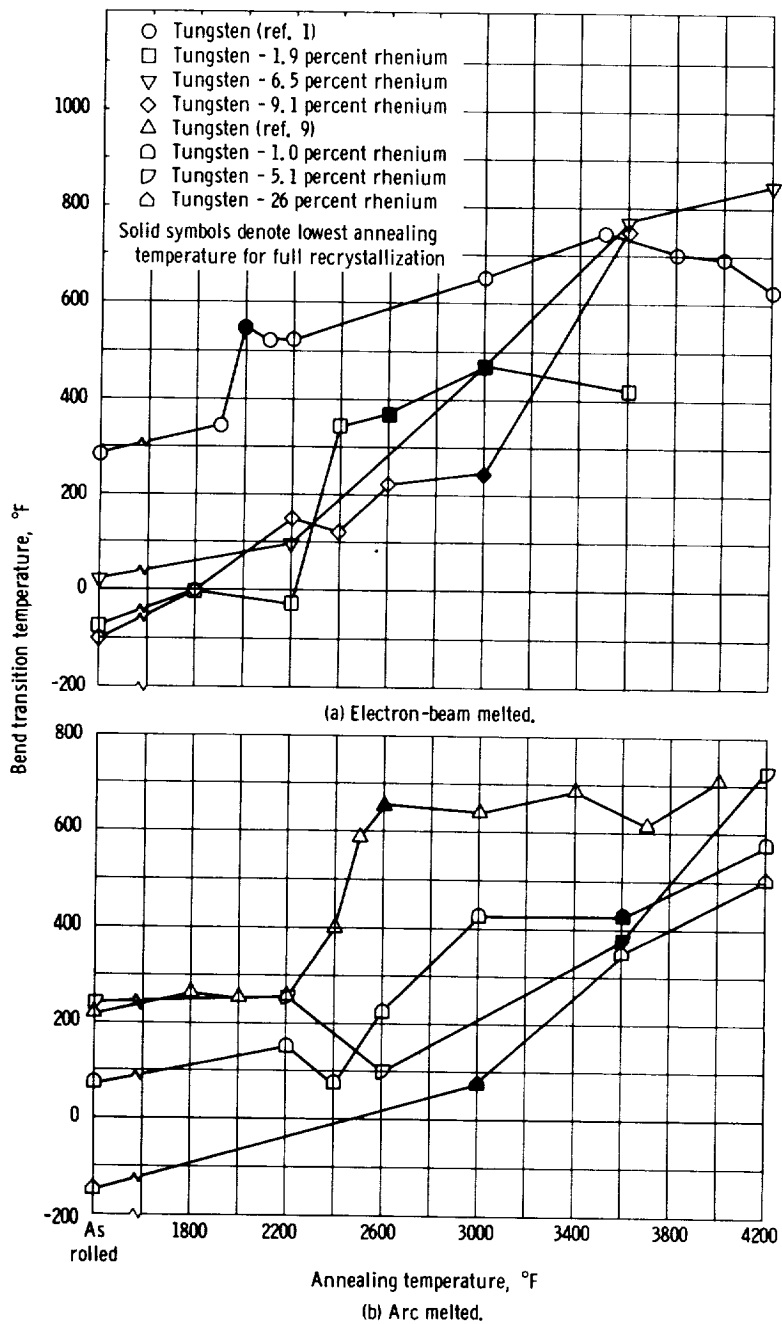


Figure 4. - Bend transition temperature of electron-beam- and arc-melted tungsten and tungsten-rhenium alloys as rolled and after annealing for 1 hour at various temperatures.

TABLE IV. - BEND TRANSITION TEMPERATURES FOR  
0.03- BY 0.5- BY 2.0-INCH SPECIMENS OF  
ELECTRON-BEAM-MELTED TUNGSTEN -  
2.8 PERCENT RHENIUM

Fabrication schedule		Bend transition temperature, °F					
Number of in-process anneals <sup>a</sup>	Finish temperature, °F	As rolled	1-Hour annealing temperature, °F				
			1800	2000	2400	2600	3600
Electropolished specimens							
0	2000	-25	---	---	25	175	400
	2200	50	---	---	50	75	450
	2400	≤75	---	---	50	225	500
	2600	≤-50	---	---	0	175	450
2	2000	25	---	---	75	125	500
As-cleaned specimens <sup>b</sup>							
0	2000	50	---	---	--	---	----
	2200	50	---	---	--	---	----
	2400	50	---	---	--	---	----
	2600	≤50	---	---	--	---	----
2	2000	150	---	---	--	---	----
	2200	50	550	650	--	---	≤475

<sup>a</sup>In-process anneals at 2200° F for 1/2 hr with sheet thicknesses of 0.070 and 0.045 in.

<sup>b</sup>Salt-bath cleaned after rolling but not electropolished.

ing at 2200° F may increase the transition temperature slightly, as compared with rolling at 2000° or lower. The 4.7-percent-rhenium alloy, however, had a much higher transition temperature, 175° F, after rolling at 2600° F than it did after rolling at 2000° F (-75° F). There was no discernible microstructural difference between the two sheets. This observation is in accord with previous studies on unalloyed tungsten (ref. 11), which indicate that the lower transition temperatures are associated with lower final rolling temperatures.

The effects of in-process stress-relief anneals of 1/2 hour at 2200° F on the electron-beam-melted tungsten - 2.8-percent-rhenium alloy were investigated. These data, which were obtained on 0.03- by 0.5- by 2.0-inch specimens, are given in table IV and indicate that there may be a slightly detrimental effect of in-process stress-relief annealing. However, the scatter in the data for the straight-rolled materials was ±50° F

and would have masked any stress-relieving effect of  $50^{\circ}$  F or less on the transition temperature.

The effects of frequent salt-bath cleaning during rolling may be significant, as seen from the data in tables II and III. The electron-beam-melted alloys with 1.9, 4.7, 9.1, and 24 percent rhenium were cleaned and conditioned three times during rolling, while the alloys with 2.5, 2.8, 4.5, and 6.5 percent rhenium were conditioned once during rolling and cleaned only after final rolling. All four of the frequently cleaned alloys exhibited exceptionally low bend transition temperatures in the as-rolled condition. These temperatures ranged from  $-75^{\circ}$  to  $-100^{\circ}$  F for the dilute alloys to approximately  $-310^{\circ}$  F for the tungsten - 24-percent-rhenium alloy. In comparison, the alloys that were conditioned only once during rolling had transition temperatures between  $25^{\circ}$  and  $-25^{\circ}$  F. Thus, the maintenance of a clean surface during rolling, which appears to reduce subsurface contamination, is quite beneficial to the subsequent ductility. This effect was not evaluated on the arc-melted alloys since initial results had shown that they have higher transition temperatures than do the electron-beam-melted alloys.

Table III also shows that the transition temperatures were generally lowest in the as-rolled condition and tended to increase slightly on stress-relief annealing after final rolling. This could have been caused by the dissolution of surface impurities during annealing.

Pack rolling was evaluated briefly on the tungsten - 1.9-percent-rhenium alloy to reduce both possible lamination associated with light reductions during final rolling and resultant inhomogeneous residual stress distributions through the sheet. Pack rolling of a single tungsten-rhenium alloy sheet from 0.06 to 0.03 inch thick between 0.06-inch molybdenum sheet at  $1800^{\circ}$  F produced an alloy sheet that was free from detectable laminations. The pack-rolled sheet, however, exhibited a transition temperature of  $175^{\circ}$  F as rolled, compared with  $-75^{\circ}$  F (table III) for the sheet straight rolled at  $2000^{\circ}$  F. Thus, although pack rolling should effect a more uniform residual stress distribution through the sheet, it appeared to raise rather than lower the ductile-brittle transition temperature under the conditions investigated. It is possible that heat losses were such that the sheet which was rolled bare at  $2000^{\circ}$  F was actually cooler than the sheet which was pack rolled at  $1800^{\circ}$  F; thus the ductility of the bare-rolled sheet was improved.

The effects of specimen size and surface preparation were evaluated on sheet of tungsten - 2.8 percent rhenium with results as given in table IV. The bend transition temperatures for electropolished specimens that measured 0.03 by 0.5 by 2.0 inches ranged from approximately  $-50^{\circ}$  to  $75^{\circ}$  F after rolling at temperatures from  $2000^{\circ}$  to  $2600^{\circ}$  F. After annealing at  $3600^{\circ}$  F, the bend transition temperatures ranged from  $400^{\circ}$  to  $500^{\circ}$  F. These transition temperatures are almost identical to those determined for 0.03- by 0.3- by 0.9-inch specimens,  $0^{\circ}$  F as rolled and  $450^{\circ}$  F after a  $3600^{\circ}$  F anneal (table III). These data indicate no detectable effect of specimen width and length in the ranges investigated.

TABLE V. - LOW-TEMPERATURE TENSILE PROPERTIES OF DILUTE TUNGSTEN-RHENIUM ALLOYS

Annealing condition	Test temperature, °F	Yield strength, psi		Ultimate tensile strength, psi	Reduction in area, percent	Elongation, percent
		Upper	Lower or 0.2-percent offset			
EB-127, tungsten - 2.5 percent rhenium, rod						
<sup>a</sup> Recrystallized	445	35 600	32 300	71 400	28	27
	530	30 600	30 500	72 400	46	31
	700	31 100	27 500	63 400	56	23
EB-139A, tungsten - 4.5 percent rhenium, rod						
As swaged	75	-----	149 500	-----	1	0.3
	280	-----	140 000	157 000	11	14
	395	-----	135 000	148 000	30	12
	495	-----	136 000	145 000	32	7
	585	-----	130 000	140 000	58	12
<sup>a</sup> Recrystallized	670	44 100	43 800	81 900	26	22
	745	39 200	38 900	75 000	51	30
	850	41 100	38 700	75 400	59	26
EB-176A, tungsten - 4.7 percent rhenium, sheet						
As rolled	75	-----	148 200	187 300	--	4
	EB-126, tungsten - 6.5 percent rhenium, rod					
<sup>a</sup> Recrystallized	600	46 900	45 100	74 500	7	----
	710	-----	53 600	80 600	45	30
	850	44 300	42 000	77 300	67	31
EB-179, tungsten - 9.1 percent rhenium, sheet						
As rolled	75	-----	227 900	250 500	--	2
	75	-----	226 000	227 500	--	3
	200	-----	222 500	224 000	--	4
	300	-----	199 800	210 500	--	5
	400	-----	202 000	206 000	--	3
	500	-----	175 800	179 500	--	3
EB-181, tungsten - 24 percent rhenium, sheet						
As rolled	75	-----	181 000	234 000	--	10
	75	-----	221 000	236 000	--	11
	200	-----	221 000	229 000	--	7

Annealing condition	Test temperature, °F	Yield strength, psi		Ultimate tensile strength, psi	Reduction in area, percent	Elongation, percent
		Upper	Lower or 0.2-percent offset			
A-132, tungsten - 1.0 percent rhenium, rod						
<sup>a</sup> Recrystallized	500	-----	-----	51 200	0	0
	550	29 600	27 300	66 800	42.6	58
	600	25 700	23 200	64 200	51.3	52
	700	17 200	16 700	60 100	52.7	50
	800	30 100	28 400	54 600	75.6	46
A-134, tungsten - 2.0 percent rhenium, rod						
<sup>a</sup> Recrystallized	400	49 600	42 600	67 800	9.2	12
	500	33 900	33 250	68 900	13.9	22
	600	32 900	30 400	66 600	52	45
	800	24 900	24 200	59 300	47.4	46
A-102, tungsten - 3.0 percent rhenium, rod						
As swaged	410	-----	105 000	124 000	54	--
	500	-----	102 000	115 000	55	--
	615	-----	105 000	113 000	56	--
<sup>a</sup> Recrystallized	380	39 000	38 100	93 800	17	--
	550	-----	34 000	81 200	44	--
	625	30 200	29 800	79 300	50	--
	800	31 500	31 000	71 200	67	--
A-99, tungsten - 5.1 percent rhenium, rod						
<sup>a</sup> Recrystallized	525	50 100	47 600	92 500	16	--
	665	57 800	46 100	82 600	53	--
	795	56 200	43 000	76 700	54	--
A-138, tungsten - 6.8 percent rhenium, rod						
<sup>a</sup> Recrystallized	300	79 700	70 200	70 200	1.2	1
	400	66 700	62 700	96 500	14	9
	600	67 400	57 500	88 700	28	20
	800	55 800	50 300	73 700	31.6	30

<sup>a</sup> Annealed in vacuum for 1 hr at 3600° F.<sup>b</sup> Average of two tests.

Data were also obtained on tungsten - 2.8-percent-rhenium specimens in the unpolished condition and are included in table IV. These specimens were cut from sheet that had been salt-bath cleaned only after final rolling. The edges were lightly deburred with emery paper prior to testing. The transition temperatures as rolled were 50° F for four of the six sheets. This suggests that electropolishing decreases the bend transition temperature, but the effect appears to be less than noted in previous work on unalloyed tungsten (refs. 12 and 13).

A substantial increase in bend transition temperature occurred when the as-cleaned material was stress-relief annealed at 1800° and 2000° F. The transition temperatures of 550° and 650° F compare with values of 75° and -25° F, respectively, for similar sheet that was electropolished to remove about 3 mils per side after it had been annealed at the same temperatures. It appears likely that surface impurities diffused into the specimen during annealing and produced a shallow case that was prone to cracking. This case can be removed by electropolishing. Care should be exercised in stress-relief annealing of sheet; the preferable method is removal of the surface layer after annealing ( e. g. , electropolishing).

### Low-Temperature Tensile Properties

Low-temperature tensile property studies were conducted on rod and sheet from six electron-beam-melted tungsten-rhenium alloys and on all five of the dilute arc-melted tungsten-rhenium alloys to determine the tensile ductile-brittle transition characteristics of these alloys. Data from these tests are presented in table V. The transition temperatures, summarized in figure 5, are based on 40 percent reduction in area, which is about half the maximum reduction in ductility observed. It is important to note that appreciable ductility is observed at temperatures as much as several hundred degrees below the nominal transition temperature, particularly on worked specimens, as seen in table V.

As seen in figure 5, the recrystallized tungsten - 2.5-percent-rhenium alloy exhibited a slightly lower tensile-transition temperature, 505° F, than did the unalloyed electron-beam-melted tungsten, 615° F (ref. 1). The transition temperatures for the arc-melted alloys that contain up to 3.0 percent rhenium range from 525° to 570° F. Limited data indicate that the as-swaged electron-beam-melted tungsten - 4.5-percent-rhenium alloy has a higher transition temperature than as-swaged unalloyed electron-beam-melted tungsten.

Room-temperature tensile data on worked sheet from several electron-beam-melted alloys indicate limited but measurable ductility. The tungsten - 4.7-percent-rhenium and tungsten - 9.1-percent-rhenium alloys showed 2 to 4 percent elongation at room temperature, while the tungsten - 24-percent-rhenium alloy showed 10 and 11 percent elongation. Although comparable data are not available on electron-beam-melted unalloyed sheet,

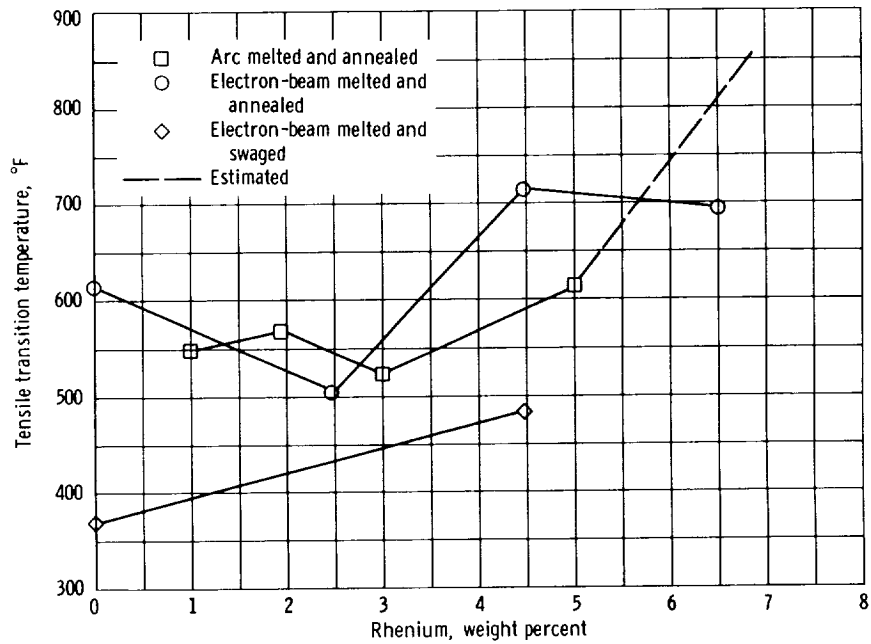


Figure 5. - Tensile transition temperature of tungsten-rhenium alloys as swaged and after annealing for 1 hour at 3600° F.

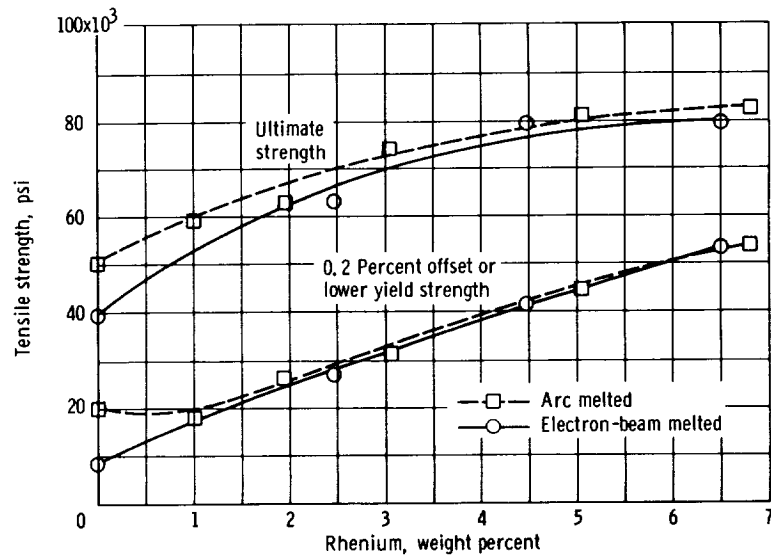


Figure 6. - Tensile strength of dilute tungsten-rhenium alloys at 700° F. (Strength values interpolated from data in table V and refs. 1 and 9.)

rod data indicate that 2 percent elongation is achieved only at about 400° F in worked specimens.

The differences in transition temperature determined in tension (fig. 5) and in bending (fig. 2, pp. 10 and 11) may reflect, at least for the worked materials, the smaller amount of work in the rod materials (68 to 81 percent) as compared with the sheet materials (89 to 94 percent). Alternatively, this may result from the difference in the amount of local strain at the defined transition temperatures, which is about 65 percent for tensile and 20 percent for bend. It would be premature, however, to ascribe the differences in ductility to tension against bending because of the differences in fabrication history and size.

The strengthening of tungsten by dilute rhenium additions at 700° F is shown in figure 6. Rhenium is a moderately effective strengthener for tungsten; it raises the ultimate tensile strength from about 50 000 to 85 000 pounds per square inch and the yield strength from about 20 000 to 50 000 pounds per square inch at the 6.5- to 6.8-percent-rhenium level.

The promotion of discontinuous yielding by dilute rhenium additions is indicated in table V. All the recrystallized alloys with 1.0 to 6.8 percent rhenium exhibited discontinuous yielding on tensile testing in the temperature range 380° to 850° F. In comparison, some but not all of the similarly evaluated specimens of unalloyed arc- and electron-beam-melted tungsten exhibited discontinuous yielding (refs. 1 and 8). The reasons for this increased tendency toward discontinuous yielding on alloying with rhenium are not clear from the present studies, but they may be the result of grain refinement on alloying. It is known that fine-grained materials are more prone to discontinuous yielding than are coarse-grained materials (ref. 14). Although the rhenium-containing materials in this study had consistently finer grain sizes than did the unalloyed materials, no tests were conducted on the grain size dependence of yielding. An alternate possibility is that rhenium may increase the tendency toward dislocation pinning by dissolved interstitials.

## High-Temperature Tensile Properties

Tensile properties were studied on both electron-beam- and arc-melted materials at 2500° to 4000° F in the worked condition and after annealing at 3600° F. Data from this study are presented in table VI. The strengths of the electron-beam-melted alloys at 3500° F are shown in figure 7 also.

Rhenium is a moderate strengthener for tungsten at these temperatures, although it is less effective than additions such as hafnium, tantalum, or columbium (ref. 6). The strength increases with increasing rhenium content to at least 9 percent, which is typical of a substitutional addition with an extensive solubility range. The improvement in



TABLE VI. - HIGH-TEMPERATURE TENSILE PROPERTIES OF DILUTE TUNGSTEN-RHENIUM ALLOYS

1-Hour annealing temperature, °F	Test temperature, °F	0.2-Percent offset yield strength, psi	Ultimate strength, psi	Elongation, percent	Reduction in area, percent	1-Hour annealing temperature, °F	Test temperature, °F	0.2-Percent offset yield strength, psi	Ultimate strength, psi	Elongation, percent	Reduction in area, percent		
EB-160, tungsten - 1.9 percent rhenium, rod						EB-181, tungsten - 24 percent rhenium, sheet							
3600	2500	14 300	28 700	62	>95	3600	2500	39 500	47 700	30	----		
	3000	-----	18 100	82			3000	25 100	28 100	63	----		
	3500	5 180	11 400	85			3500	14 000	14 000	110	----		
	4000	4 010	6 550	112			4000	6 310	6 310	143	----		
EB-127, tungsten - 2.5 percent rhenium, rod						A-132, tungsten - 1.0 percent rhenium, rod							
As swaged	2500	39 500	56 300	12	(a)	As swaged	3000	33 300	34 300	23	>98		
	3000	16 800	24 100	31	(a)		3500	4 800	11 900	62	>98		
	3500	4 720	11 200	80	>95		4000	4 800	7 100	86	>98		
3600	2500	14 800	30 800	41	(a)	3600	2500	14 000	28 100	64	>98		
	3000	9 160	21 800	50	>95		3000	9 400	18 100	78	>98		
	3500	4 810	12 300	78	>95		3500	6 300	11 300	83	>98		
	4000	3 490	7 840	105	>95								
EB-156, tungsten - 2.8 percent rhenium, sheet						A-134, tungsten - 2.0 percent rhenium, rod							
As rolled	2500	51 200	62 600	9	----	As swaged	3000	35 300	35 500	35	>98		
	3000	15 100	20 800	48	----		3500	9 800	13 300	68	>98		
	3500	6 880	11 400	13	----		4000	5 700	8 300	76	>98		
3600	2500	10 100	24 700	32	----	3600	2500	14 700	28 700	73	>98		
	3000	8 560	17 100	42	----		3000	9 800	18 700	51	>98		
	3500	6 300	11 400	62	----		3500	8 030	12 500	96	>98		
EB-159, tungsten - 3.6 percent rhenium, rod						A-102, tungsten - 3.0 percent rhenium, rod							
3600	2500	16 900	34 400	56	>95	As swaged	2500	54 200	63 000	17	95		
	3000	11 300	20 800	77	>95		3000	35 400	45 300	26	95		
	3500	7 040	12 800	77	>95		3500	6 500	13 900	92	>98		
	4000	4 560	7 590	130	>95	3600	2500	14 500	37 100	47	>98		
EB-139, tungsten - 4.5 percent rhenium, rod							3000	11 000	23 700	71	>98		
As swaged	2500	49 000	69 200	20	91		3500	6 260	13 100	84	95		
	3000	34 200	36 500	38	>95		4000	4 240	8 130	132	98		
	3500	9 330	12 900	106	>95	4200	3500	5 650	12 500	85	83		
3600	2500	18 500	36 700	68	93								
	3000	13 600	23 500	73	>95								
	3500	8 390	13 700	102	>95								
	4000	5 180	7 130	98	>95								
EB-176, tungsten - 4.7 percent rhenium, sheet						A-99, tungsten - 5.1 percent rhenium, rod							
As rolled	3000	29 900	31 200	29	----	As swaged	2500	66 700	112 000	17	85		
	3500	7 810	11 600	41	----		3000	46 800	64 500	20	94		
3600	2500	21 200	42 300	48	>95		3600	2500	21 400	38 600	---	----	
	3000	11 200	22 900	82	93	3000		19 900	35 000	59	97		
	3500	7 010	14 600	94	>95	3500		7 200	14 800	89	>98		
	4200	3 220	7 140	89	>95	4000		3 760	8 840	87	73		
EB-126, tungsten - 6.5 percent rhenium, rod						A-138, tungsten - 6.8 percent rhenium, rod							
As swaged	2500	51 500	73 300	23	52	As swaged	3000	-----	52 600	23	55		
	3000	29 000	38 000	24	45		3500	17 600	19 900	22	47		
	3500	6 920	13 600	79	>95	3600	2500	-----	46 800	49	50		
3600	2500	21 200	42 300	48	>95		3000	25 700	30 200	30	51		
	3000	11 200	22 900	82	93		3500	15 300	17 400	56	----		
	3500	7 010	14 600	94	>95	Tungsten - 26 percent rhenium, sheet							
	4200	3 220	7 140	89	>95	As received	2500	48 500	63 400	---	----		
EB-179, tungsten - 9.1 percent rhenium, sheet							3600	2500	40 600	49 100	53	----	
3600	2500	23 700	35 800	35	----			3000	26 900	29 700	72	----	
	3000	16 300	23 500	59	----			3500	12 600	13 100	119	----	
	3500	8 820	12 900	41	----								
	4000	6 930	8 010	87	----								

<sup>a</sup>Specimen split.

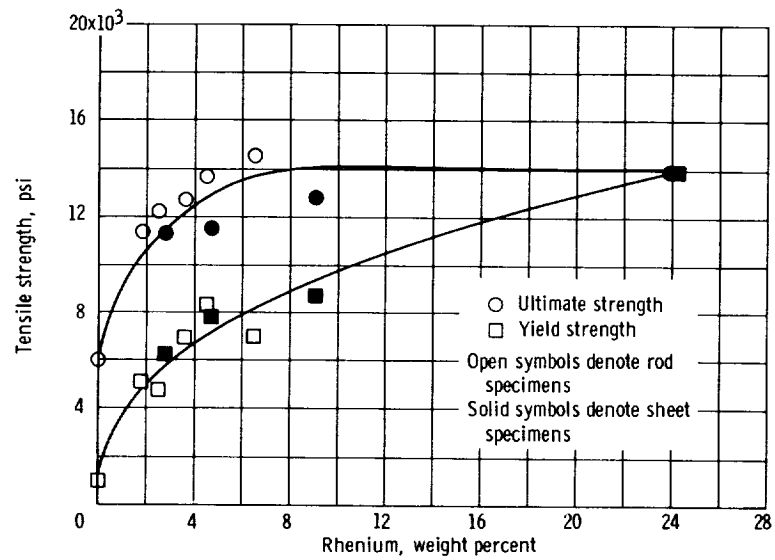


Figure 7. - Tensile strength of annealed electron-beam-melted tungsten-rhenium alloys at 3500° F.

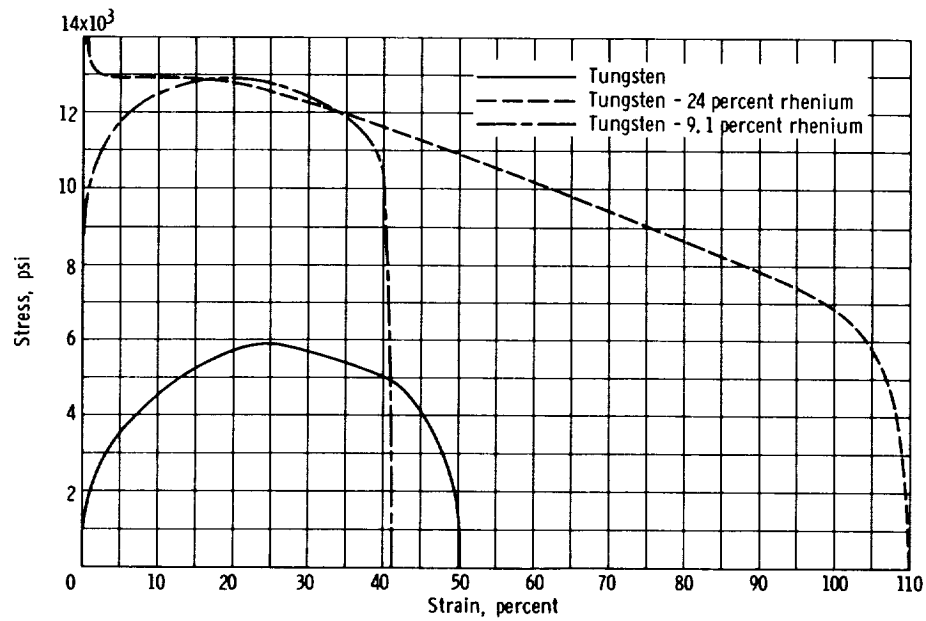


Figure 8. - Engineering stress-strain curves for electron-beam-melted unalloyed tungsten, tungsten - 9.1 percent rhenium, and tungsten - 24 percent rhenium sheet. Temperature, 3500° F; strain rate, 0.05 minute<sup>-1</sup>.

ultimate tensile strength at 3500<sup>0</sup> F is about 130 percent for electron-beam-melted alloys with 6 percent or more rhenium (fig. 7). The electron-beam-melted alloys are 2000 to 3000 pounds per square inch weaker than are the arc-melted alloys at 3500<sup>0</sup> F, which reflects the larger grain size and higher purities of the electron-beam-melted alloys. Rhenium also confers a similar strength improvement at lower temperatures. The improvement at 2500<sup>0</sup> F, for example, is approximately 150 percent.

The high-rhenium alloys exhibit several unusual deformation characteristics at elevated temperatures. As shown by table VI, the elongations of the electron-beam-melted tungsten - 24-percent-rhenium alloy and the arc-melted tungsten - 26-percent-rhenium alloy are higher than those of the dilute alloys and increase substantially with increasing temperature.

Another significant deformation characteristic is the low work hardening in the high-rhenium alloys, as illustrated in figure 8. Unalloyed tungsten and tungsten - 9.1 percent rhenium exhibit normal stress-strain curves, while the tungsten - 24-percent-rhenium alloy exhibits a sharp decrease in stress (about 8 percent) immediately after yielding and shows no evidence of subsequent work hardening.

These observations, together with the fact that the tungsten - 24-percent-rhenium and tungsten - 26-percent-rhenium alloys are close to the solubility limit for rhenium in tungsten, suggest that this behavior may be related to a strain-induced structural change in the alloy. A more complete study of the unusual behavior of these alloys, however, was beyond the scope of this report.

## High-Temperature Creep Behavior

The creep behavior of electron-beam- and arc-melted tungsten-rhenium alloys was studied by step-load creep tests at 3000<sup>0</sup> and 3500<sup>0</sup> F. The results are presented in tables VII and VIII and summarized in figures 9 and 10.

The creep behavior of the alloys is similar to that of unalloyed tungsten (ref. 8) in that both transient and steady creep were observed at 3000<sup>0</sup>, while steady creep was the primary observation at 3500<sup>0</sup> F.

The log-log plots of stress against steady creep rate at 3500<sup>0</sup> F gave essentially straight line relations (fig. 9), which indicates that the creep rate is a power function of stress, for example,

$$\dot{\epsilon} = K\sigma^n$$

where

TABLE VII. - CREEP PROPERTIES OF ELECTRON-BEAM-MELTED TUNGSTEN-RHENIUM ALLOYS

Alloy	Analyzed rhenium content, wt percent (a)	Test temperature, °F (b)	Stress, psi	Steady creep rate, sec <sup>-1</sup>	Stress factor, n	Alloy	Analyzed rhenium content, wt percent (a)	Test temperature, °F (b)	Stress, psi	Steady creep rate, sec <sup>-1</sup>	Stress factor, n
EB-160A	1.9	3500	2380 2860 3460 4170	0.10×10 <sup>6</sup> .28 .76 1.6	5.1	EB-176	4.7	3500	3540 4190 4830 5470 6110	0.50×10 <sup>6</sup> 1.3 2.7 5.8 9.1	5.3
EB-127	2.5	3000	9030 9760	5.2×10 <sup>6</sup> 6.4	---	EB-126	6.5	3500	2620 3810 4400 5000 5950	0.19×10 <sup>6</sup> 1.8 1.9 3.3 7.4	4.4
		3500	3000 3350 4070 4910 5990	0.44×10 <sup>6</sup> 1.1 2.4 6.6 16	5.0			<sup>c</sup> 3500	2610 3560 4510 5220 6170 7120	0.15×10 <sup>6</sup> .71 2.3 3.8 7.4 12	
EB-156	2.8 (0.03-in. sheet)	3000 (As rolled) 3500 (As rolled)	8790 2750 3670 4580 5500 6410	1.8×10 <sup>6</sup> .067 .45 1.6 4.4 9.8	5.6	EB-179	9.1	3500	2660 3320 3980 4650 5310 6640	0.10×10 <sup>6</sup> .33 .80 2.0 3.8 11	5.3
		3500	3490 4180 4880 5930	0.51×10 <sup>6</sup> 1.2 3.5 9.1	5.7	EB-181	24	3500	2970 3720 4460 5200	1.5×10 <sup>6</sup> 3.5 7.7 14	4.0
EB-159A	3.6	3500	3050 3690 4450 5340	0.29×10 <sup>6</sup> .78 1.9 3.4	4.4						
EB-139	4.5	3000	4470 5340 6450 7700 9180	0.073×10 <sup>6</sup> .11 .26 .58 1.4	4.5						
		3500	2130 2510 2880 3380 4000 4760 5510	0.078×10 <sup>6</sup> .12 .17 .33 .84 1.8 3.3	4.7						

<sup>a</sup>Rod specimens with 0.16-in. -diam reduced section employed except where sheet is indicated.<sup>b</sup>Specimens annealed in vacuum for 1 hr at 3600° F except where indicated.<sup>c</sup>Duplicate test.

TABLE VIII. - CREEP PROPERTIES OF ARC-MELTED TUNGSTEN-RHENIUM ALLOYS

Alloy	Analyzed rhenium content, wt percent (a)	Test tempera- ture, °F (b)	Stress, psi	Steady creep rate, sec <sup>-1</sup>	Stress factor, n	Alloy	Analyzed rhenium content, wt percent (a)	Test tempera- ture, °F (b)	Stress, psi	Steady creep rate, sec <sup>-1</sup>	Stress factor, n
A-132	1.0	3500	2 620 2 860 3 450 4 160 5 000 5 480	0.22×10 <sup>6</sup> .32 .58 1.5 4.1 6.1	4.6	A-99	5.1	3000	6 890	0.16×10 <sup>6</sup>	---
A-134	2.0	3500	2 920 3 640 4 370 5 220 6 190	0.058×10 <sup>6</sup> .45 1.5 4.7 12	6.1	A-138	6.8	3500	3 450 4 170 5 240 6 430 7 740	0.10×10 <sup>6</sup> .20 .55 1.9 5.6	5.2 5.5 5.5
A-102	3.0	3000	6 650 7 390 8 370 9 360 10 500	0.32×10 <sup>6</sup> .65 1.3 2.7 3.6	5.5	----- (Sheet)	26	3000	5 870 7 170 8 470 10 100 12 100	0.29×10 <sup>6</sup> .75 2.1 5.2 15	5.0
		3500	4 010 4 750 5 110 6 210	1.0×10 <sup>6</sup> 2.9 4.7 14	6.3			3500	2 560 3 210 3 850 4 490 5 130 5 770	0.47×10 <sup>6</sup> .77 1.8 3.5 6.9 13	4.7
		c3500	3 970 4 710 5 090 6 200	0.78×10 <sup>6</sup> 2.2 3.7 12	6.3						

<sup>a</sup> Rod specimens with 0.16-in. -diam reduced section employed except where sheet is indicated.<sup>b</sup> All specimens vacuum annealed for 1 hr at 3600° F prior to testing except where indicated.<sup>c</sup> Annealed for 1 hr in vacuum at 4000° F prior to testing.

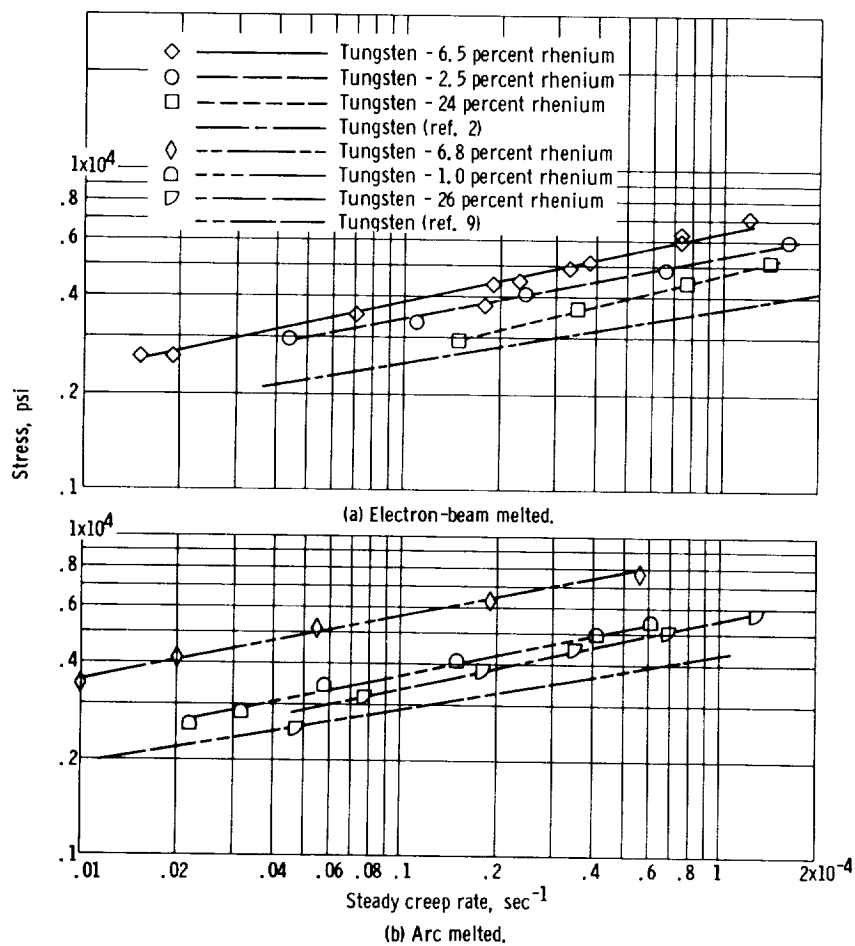


Figure 9. - Representative creep rate data for electron beam- and arc-melted tungsten and tungsten-rhenium alloys at 3500° F.

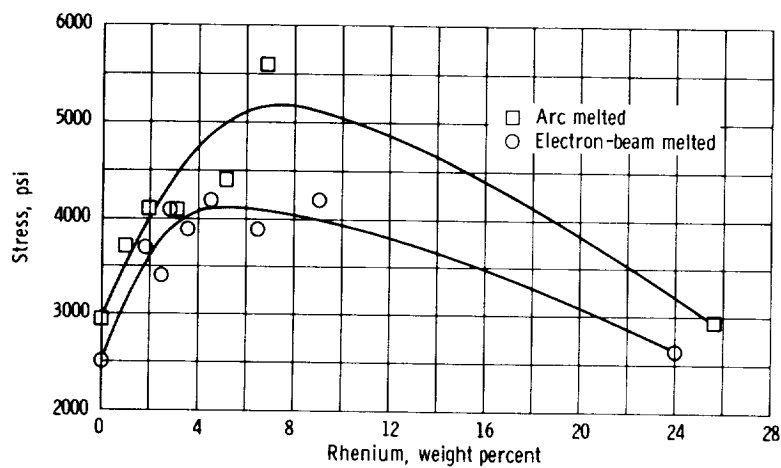


Figure 10. - Effect of rhenium on strength of tungsten-rhenium alloys. Steady creep rate, 10<sup>-6</sup> per second; temperature, 3500° F (corresponds approximately to rupture life of 50 hrs).

- $\dot{\epsilon}$  steady creep rate,  $\text{sec}^{-1}$   
 K temperature-dependent constant  
 $\sigma$  stress based on initial cross section, psi  
 n stress factor

The constant  $n$  has been determined as 5.8 for unalloyed electron-beam- and arc-melted tungsten. Values of  $n$  for the electron-beam-melted tungsten-rhenium alloys were slightly lower, from 4.0 to 5.7, while  $n$  ranged from 4.6 to 6.1 for the arc-melted alloys. It has previously been observed that alloys generally exhibit lower values of  $n$  than do the unalloyed solvent metals (ref. 15).

The strength at a steady creep rate of  $10^{-6}$  per second was interpolated from stress-creep-rate plots and is shown in figure 10 as a function of rhenium content. This creep rate corresponds to a rupture life of approximately 50 hours. Additions of rhenium up to 6 to 8 percent increase the creep strength of tungsten by about 70 percent, approximately half the increase observed in short-time tensile testing. The high-rhenium alloys with 24 and 26 percent rhenium, however, are considerably weaker than the dilute alloys, since the high-rhenium alloys have approximately the same strength as unalloyed tungsten at  $3500^{\circ}\text{F}$ . This is in contrast to the behavior during short-time tensile testing, where the high-rhenium alloys had almost the same strength as the dilute alloys. (Both dilute and high-rhenium alloys are considerably stronger than unalloyed tungsten.) This behavior suggests that the dislocation climb, which is assumed to control the creep rate at these temperatures, is more rapid in these alloys than in the stronger dilute tungsten-rhenium alloys.

## Recrystallization and Grain Growth Behavior

Recrystallization and grain growth behavior of electron-beam-melted alloys were studied on both rod and sheet. The pertinent metallographic features after 1-hour heat treatments at  $2400^{\circ}$  to  $4000^{\circ}\text{F}$  are summarized in table IX. The recrystallization temperature (50 percent recrystallized in 1 hr) and the grain size after annealing at  $3600^{\circ}\text{F}$  are summarized in figures 11 and 12.

These data indicate that rhenium significantly increases the recrystallization temperature of tungsten, even at alloying levels as low as 1.9 percent. As shown in figure 11, the tungsten - 1.9-percent-rhenium alloy has a recrystallization temperature of  $2790^{\circ}\text{F}$  in rod form, which is an increase of  $590^{\circ}\text{F}$  over the recrystallization temperature of unalloyed tungsten. Increasing the rhenium content to 6.5 percent raises the recrystallization temperature to  $2950^{\circ}\text{F}$ , which is  $750^{\circ}\text{F}$  more than that of unalloyed tungsten.

TABLE IX. - RECRYSTALLIZATION AND GRAIN GROWTH OF ELECTRON-BEAM-MELTED TUNGSTEN-RHENIUM ALLOYS

Shape	Prior reduc- tion, percent	Annealing tempera- ture, °F (a)	Fraction recrystal- lized	Average grain diameter, in.	Shape	Prior reduc- tion, percent	Annealing tempera- ture, °F (a)	Fraction recrystal- lized	Average grain diameter, in.					
EB-160A, tungsten - 1.9 percent rhenium					EB-139B, tungsten - 4.5 percent rhenium									
0.35-in. rod	76	2400	0	-----	0.03-in. sheet (rolled at 1800° F)	91	2700	0.10	-----					
		2500	0	-----			2800	.80	-----					
		2600	.20	-----			2900	1.00	-----					
		2700	.31	-----	0.03-in. sheet (rolled at 2200° F)	91	2700	0.02	-----					
		2800	.51	-----			2800	.50	-----					
		2900	.73	-----			2900	1.00	-----					
		3000	1.00	0.0010			EB-126, tungsten - 6.5 percent rhenium							
		3100	1.00	.0014			0.35-in. rod	68	2600	0	-----			
3200	1.00	.0015	2800	.45	-----									
EB-127, tungsten - 2.5 percent rhenium					3000	.39			-----					
0.35-in. rod	76	2600	0.06	-----	3100	.31			-----					
		2800	.18	-----	3200	.88			-----					
		2900	.16	-----	3300	1.00			0.0014					
		3000	.95	-----	3400	↓			.0019					
		3100	1.00	0.0012	3600	↓			c.0028					
		3200	↓	.0010	3800	↓			.0043					
		3400	↓	.0022	4000	↓			.0075					
		3600	↓	b.0033	0.05-in. sheet		2600	0	-----					
3800	↓	.0047	2800	.19			-----							
4000	↓	.0087	3000	.87			-----							
EB-156, tungsten - 2.8 percent rhenium							3100	.79	-----					
0.05-in. sheet	94	2600	0.28	-----	3200	1.00	0.0010	EB-179, tungsten - 9.1 percent rhenium						
		2800	.63	-----	0.03-in. sheet	93	2400	0	-----					
		3000	1.00	0.0011			2500	.02	-----					
		3600	1.00	.0034			2700	.10	-----					
EB-159A, tungsten - 3.6 percent rhenium							2800	.80	-----					
0.03-in. sheet (rolled at 1800° F)	91	2700	0.10	-----			2900	1.00	0.00071	0.03-in. sheet		2400	0	-----
		2800	.80	-----			3000	↓	.00083					
		2900	1.00	-----			3100	↓	.00071					
		0.03-in. sheet (rolled at 2200° F)	91	2700			0.02	-----	3200			↓	.00095	
				2800			.50	-----	3300			↓	.0011	
2900	1.00			-----			3400	↓	.0014					
0.03-in. sheet (rolled at 2200° F with two anneals)	91			2500			0	-----	3600			↓	.0020	
				2600	.02	-----	EB-181, tungsten - 24 percent rhenium							
		2700	.50	-----	0.03-in. sheet		2400	0	-----					
		2800	.67	-----			2500	.05	-----					
		2900	1.00	-----			2700	.60	-----					
EB-159A, tungsten - 3.6 percent rhenium							2800	.80	-----					
0.35-in. rod	76	2700	0	-----			2900	1.00	0.00059					
		2800	.01	-----			3000	↓	.00095					
		2900	.48	-----			3100	↓	.0012					
		3000	.94	0.00071			3200	↓	.0014					
		3100	1.00	.00098			3300	↓	.0014					
		3200	1.00	.0011			3400	↓	.0020					
							3600	↓	.0026					

<sup>a</sup> Annealing time, 1 hr.<sup>b</sup> Average of three specimens.<sup>c</sup> Average of two specimens.



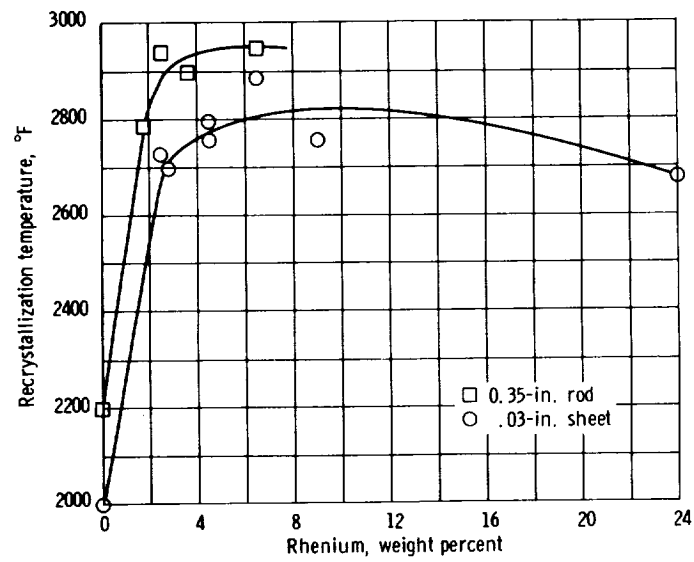


Figure 11. - Temperature for 50 percent recrystallization in 1 hour for electron-beam-melted tungsten-rhenium alloys.

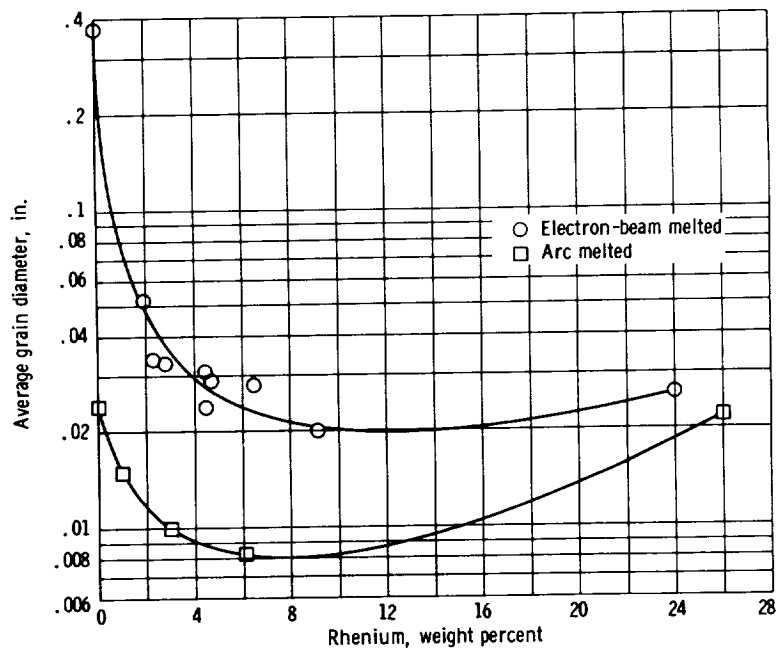


Figure 12. - Effect of rhenium content on grain size of tungsten sheet after annealing for 1 hour at 3600° F. (Grain sizes for unalloyed tungsten estimated from data in refs. 2 and 9.)

Sheet data indicate a similar sharp increase in the recrystallization temperature on alloying with rhenium. The tungsten - 24-percent-rhenium alloy recrystallizes at  $2680^{\circ}\text{F}$ , which is lower than the  $2890^{\circ}\text{F}$  observed for the tungsten - 6.5-percent-rhenium alloy but substantially higher than the  $2000^{\circ}\text{F}$  determined for unalloyed tungsten. Although no recrystallization studies were conducted on arc-melted alloys, they would be expected to recrystallize several hundred degrees higher than the electron-beam-melted alloys. Unalloyed arc-melted tungsten recrystallizes at about  $2700^{\circ}\text{F}$  (ref. 8), about  $500^{\circ}\text{F}$  higher than electron-beam-melted tungsten.

The effect of rhenium on grain growth after recrystallization is illustrated in figure 12, which is a plot of the grain size after annealing at  $3600^{\circ}\text{F}$ . As with recrystallization, dilute rhenium alloying is very effective in grain refining. The addition of about 3 percent rhenium decreases the grain size of electron-beam-melted tungsten sheet by about one order of magnitude. A minimum in both the grain size and the associated grain growth rates occurs somewhere between about 6 and 24 percent rhenium. This behavior is analogous to the recrystallization behavior because the low-rhenium alloys have the highest recrystallization temperatures and the smallest annealed grain sizes, while the high-rhenium alloy (24 percent) has a slightly lower recrystallization temperature and a slightly larger grain size.

The arc-melted alloys were finer grained than corresponding electron-beam-melted alloys because of the lower purities of the arc-melted alloys. The behavior of the arc-melted alloys was similar to that of the electron-beam-melted alloys in that a minimum in grain size is indicated in the range between 6 and 26 percent rhenium.

## SUMMARY OF RESULTS

Electron-beam- and arc-melted tungsten and tungsten alloys were evaluated for ductility, strength, recrystallization, and grain growth characteristics. The following observations were made:

1. Sheet fabricated from electron-beam-melted tungsten alloys with 1.9 to 9.1 percent rhenium exhibited ductile-brittle bend transition temperatures in the worked condition as low as  $-75^{\circ}\text{F}$  to  $-100^{\circ}\text{F}$ , as compared with  $235^{\circ}\text{F}$  for unalloyed tungsten (electropolished). Sheet fabricated from arc-melted alloys was less ductile, with bend transition temperatures of  $50^{\circ}\text{F}$  to  $280^{\circ}\text{F}$ . This difference suggests that the improved ductility may be related in part to the higher purity achieved by electron-beam melting.

2. Important fabrication variables include cleanliness during rolling and rolling temperature. The best ductilities were obtained on sheet that was cleaned several times during rolling at temperatures of  $1750^{\circ}\text{F}$  to  $2400^{\circ}\text{F}$ . Stress-relief annealing during or after rolling had little effect on ductility.

3. Annealing at 3600<sup>0</sup> F significantly increased the ductile-brittle bend transition temperatures of both electron-beam- and arc-melted alloys. Transition temperatures of about 400<sup>0</sup> F were observed for alloys with 2 to 4 percent rhenium. High-rhenium alloys with 24 and 26 percent rhenium had transition temperatures of 350<sup>0</sup> to 375<sup>0</sup> F after similar annealing treatments.

4. Alloying with 1.9 to 9.1 percent rhenium raised the recrystallization temperature of the electron-beam-melted tungsten by 600<sup>0</sup> to 800<sup>0</sup> F. The grain growth rates were significantly reduced.

5. Rhenium additions up to 9.1 percent strengthened tungsten at elevated temperatures in both short-time tensile and long-time creep tests. The alloys with 24 and 26 percent rhenium had tensile strengths similar to the 9.1-percent-rhenium alloy, but they were considerably weaker in creep; strengths approximated those of unalloyed tungsten.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, March 31, 1966.

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